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# Dichlorido[tris(1*H*-benzimidazol-2-ylmethyl)amine-$\kappa^4N,N^3,N^{3'},N^{3''}$]iron(III) chloride tetrahydrofuran monosolvate monohydrate

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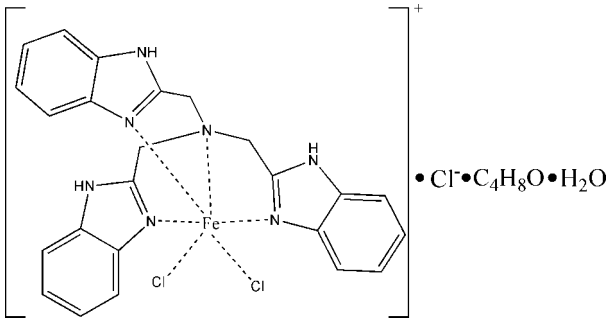
Key indicators: single-crystal X-ray study; $T$ = 292 K; mean $\sigma$(C–C) = 0.005 Å; disorder in solvent or counterion; $R$ factor = 0.047; $wR$ factor = 0.116; data-to-parameter ratio = 13.7.

In the title compound, $[FeCl_2(C_{24}H_{21}N_7)]Cl\cdot C_4H_8O\cdot H_2O$, the $Fe^{III}$ atom is coordinated by four N atoms of the polybenzimidazole ligand and two Cl atoms in a distorted octahedral environment. The cation, anion, the uncoordinated water molecule and the THF solvent molecule are linked by hydrogen bonds into a three-dimensional network structure. The THF molecule is disordered with two sets of sites in a 0.58 (1):0.42 (2) ratio..

## Related literature

For the synthesis of the ligand, see: Hendriks *et al.* (1982). For benzimidazole-like ligands, see: Moon & Soo Lah (2002).




## Experimental

### Crystal data

$[FeCl_2(C_{24}H_{21}N_7)]Cl\cdot C_4H_8O\cdot H_2O$
$M_r$ = 659.80
Monoclinic, $P2_1/c$
$a$ = 10.2898 (4) Å
$b$ = 13.7475 (5) Å
$c$ = 21.5271 (7) Å
$\beta$ = 101.614 (1)°
$V$ = 2982.85 (19) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.81 mm$^{-1}$
$T$ = 292 K
0.20 × 0.15 × 0.10 mm

### Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min}$ = 0.844, $T_{max}$ = 0.923
17337 measured reflections
5527 independent reflections
3999 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.046

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.116
$S$ = 1.00
5527 reflections
404 parameters
21 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.33 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.25 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1$W$—H1$A$···Cl3 | 0.828 (18) | 2.34 (2) | 3.136 (3) | 160 (4) |
| O1$W$—H1$B$···Cl1 | 0.831 (19) | 2.51 (3) | 3.267 (3) | 152 (4) |
| N2—H2···Cl3$^{i}$ | 0.859 (18) | 2.25 (2) | 3.070 (3) | 159 (3) |
| N4—H4···O1$W^{ii}$ | 0.862 (18) | 1.96 (2) | 2.795 (4) | 162 (3) |
| N6—H6···O1$W^{iii}$ | 0.852 (18) | 1.91 (2) | 2.741 (4) | 163 (3) |

Symmetry codes: (i) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (iii) $x+1, -y+\frac{1}{2}, z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2597).

## References

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

# supporting information

*Acta Cryst.* (2009). E**65**, m828 [doi:10.1107/S1600536809023423]

# Dichlorido[tris(1*H*-benzimidazol-2-ylmethyl)amine-$\kappa^4N,N^3,N^{3'},N^{3''}$]iron(III) chloride tetrahydrofuran monosolvate monohydrate

## Dong'e Wang

### S1. Comment

Tris(1-*H*-benzimidazol-2-ylmethl)amine-N, $H_3$ntb, is a typical heterocyclic ligand with nitrogen as the donor atom. Over the past decades, there has been an interest in the $Fe^{III}$ ions coordinated to benzimidazole-rich ligands in particular with respect to relationships between the activity and structural properties of metalloenzymes such as superoxide dismutases (SOD) (Moon & Soo Lah, 2002). Here, we report our findings in the title compound (I).

In compound (I) (Fig.1), the asymmetric unit consists of one $[Fe(H_3ntb).Cl_2]^+$ cation, one $Cl^-$ anion and each one water and tetrahydrofuran molecules. The $Fe^{III}$ is coordinated by four nitrogen atoms of a tetradentate liangd ntb, and two chloride anions, forming a distorted octahedron. The Fe—$N_{amine}$ bond length (2.355 (2) Å) is more longer than the mean bond length of Fe—$N_{benzimidazole}$ (2.101 (2) Å), which is owing to the steric requirement. No other abnormal bond lengths and bond angles are observed in (I).

In the crystal, the ions are joined together by extensive hydrogen bondings and and $\pi$-$\pi$ interactions, which lead to the formation of a three dimensional network (Fig.2).

### S2. Experimental

All reagants and solvents were used as obtained without further purification. $H_3$ntb was synthesized according to the literature (Hendriks *et al.*,1982). A mixture of $FeCl_3$ (0.0324 g, 0.2 mmol), $H_3$ntb(0.0814 g, 0.2 mmol), tetrahydrofuran (5 ml)was transferred to and sealed in a Parr Teflon-lined stainless steel vessel (15 ml), which was heated at 393 K for 3 days. After cooling to room temperature, the red block crystals of I were filtered off, washed with distilled water, and dried at ambient temperature (39 mg, yield 28.7% based on $FeCl_3$).

### S3. Refinement

During the refinement, C27 and C28 atoms were founded to be disordered over two positions and their occupancies were refined by using ′PART′ command and some C–O and C–C distance of the THF molecule were refined by using command of ′DFIX′. The final satisfactory outcome was 0.58 (1):0.45 (2) for the major and minor components, respectively. H atoms bonded to carbon atoms were located at the geometrical positions with C—H=0.93Å (aromatic), 0.97 Å (methylene) and $U_{iso}$(H) =1.2$U_{eq}$(C) (aromatic and methlene). H atoms bonded N and O atoms were found from the difference maps with the constraints of N—H=0.86 (1) Å, O—H=0.82 (1) Å and H—H=1.35 (1) Å, with the thermal factors being set k times of their carrier atoms (k=1.2 for N and 1.5 for O atoms).

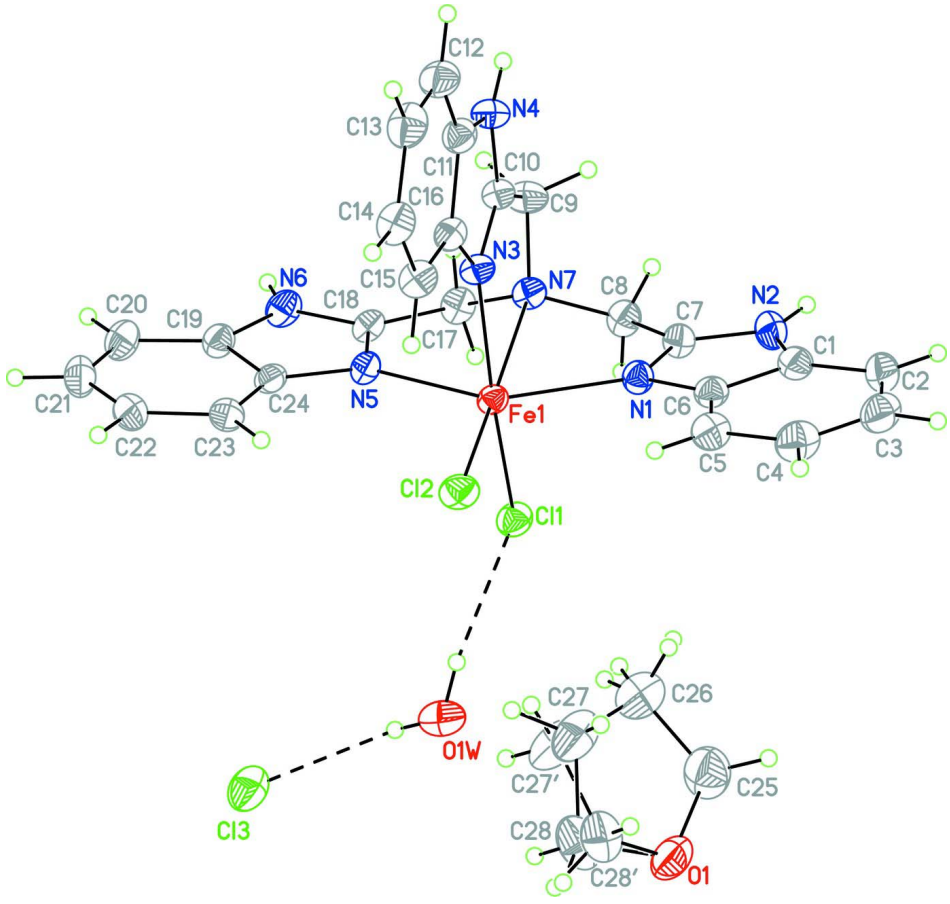


**Figure 1**

A view of the structure of (I), with displacement ellipsoids drawn at the 30% probability level. Hydrogen bonds are shown as dashed lines.

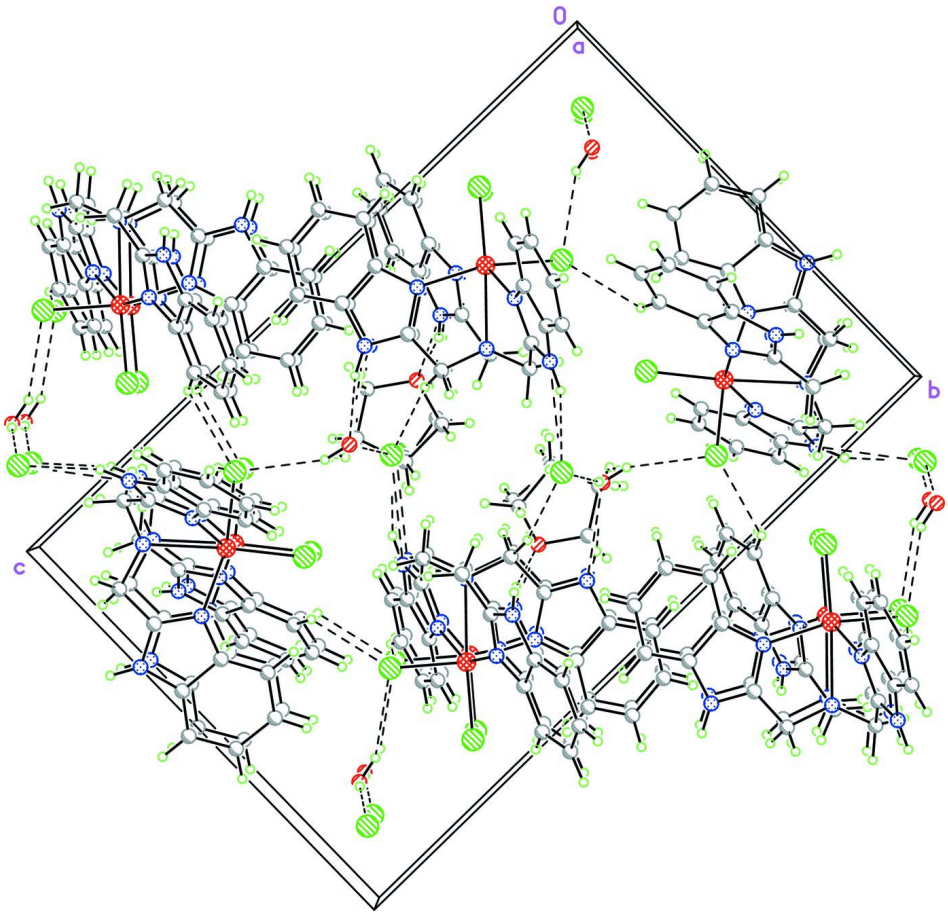


**Figure 2**

Plot of the crystal packing showing the linkage of the molecules by H-bonding shown as dashed lines.

## Dichlorido[tris(1*H*-benzimidazol-2-ylmethyl)amine-$\kappa^4N,N^3,N^{3'},N^{3''}$]iron(III) chloride tetrahydrofuran monosolvate monohydrate

*Crystal data*

$[FeCl_2(C_{24}H_{21}N_7)]Cl\cdot C_4H_8O\cdot H_2O$
$M_r = 659.80$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 10.2898\ (4)$ Å
$b = 13.7475\ (5)$ Å
$c = 21.5271\ (7)$ Å
$\beta = 101.614\ (1)°$
$V = 2982.85\ (19)$ Å$^3$
$Z = 4$
$F(000) = 1364$
$D_x = 1.469$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3638 reflections
$\theta = 2.4–22.2°$
$\mu = 0.81$ mm$^{-1}$
$T = 292$ K
Block, red
0.20 × 0.15 × 0.10 mm

*Data collection*

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine focus sealed Siemens Mo tube
Graphite monochromator
0.3° wide $\omega$ exposures scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{min} = 0.844$, $T_{max} = 0.923$
17337 measured reflections

5527 independent reflections
3999 reflections with $I > 2\sigma(I)$
$R_{int} = 0.046$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.4°$
$h = -12 \rightarrow 12$
$k = -16 \rightarrow 15$
$l = -26 \rightarrow 24$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.116$
$S = 1.00$
5527 reflections
404 parameters
21 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å$^{-3}$
$\Delta\rho_{min} = -0.25$ e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on $F^2$, conventional R-factors R are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2$sigma($F^2$) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on $F^2$ are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Fe1 | 0.94620 (4) | 0.27332 (3) | 0.810133 (19) | 0.03211 (14) | |
| Cl1 | 1.00873 (8) | 0.17478 (6) | 0.73359 (4) | 0.0445 (2) | |
| Cl2 | 0.86507 (8) | 0.39490 (6) | 0.74462 (4) | 0.0476 (2) | |
| N1 | 0.7813 (2) | 0.18448 (19) | 0.81255 (12) | 0.0369 (6) | |
| N2 | 0.6965 (3) | 0.0441 (2) | 0.83683 (14) | 0.0482 (7) | |
| H2 | 0.694 (3) | −0.0126 (16) | 0.8535 (15) | 0.058* | |
| N3 | 0.9114 (2) | 0.33689 (18) | 0.89433 (11) | 0.0367 (6) | |
| N4 | 0.9099 (3) | 0.3256 (2) | 0.99701 (12) | 0.0439 (7) | |
| H4 | 0.924 (3) | 0.296 (2) | 1.0330 (11) | 0.053* | |
| N5 | 1.1468 (2) | 0.30502 (18) | 0.84551 (11) | 0.0342 (6) | |
| N6 | 1.3466 (2) | 0.2668 (2) | 0.89915 (13) | 0.0446 (7) | |
| H6 | 1.413 (2) | 0.237 (2) | 0.9213 (14) | 0.054* | |
| N7 | 1.0223 (2) | 0.15132 (18) | 0.88547 (11) | 0.0361 (6) | |
| C1 | 0.5896 (3) | 0.0998 (3) | 0.80784 (15) | 0.0452 (8) | |
| C2 | 0.4545 (3) | 0.0799 (3) | 0.79198 (18) | 0.0604 (10) | |
| H2A | 0.4191 | 0.0210 | 0.8019 | 0.072* | |
| C3 | 0.3760 (4) | 0.1536 (3) | 0.76056 (18) | 0.0635 (11) | |
| H3 | 0.2851 | 0.1433 | 0.7487 | 0.076* | |
| C4 | 0.4273 (3) | 0.2419 (3) | 0.74607 (18) | 0.0596 (10) | |
| H4A | 0.3703 | 0.2893 | 0.7251 | 0.071* | |

| | | | | | |
|---|---|---|---|---|---|
| C5 | 0.5621 (3) | 0.2610 (3) | 0.76226 (16) | 0.0508 (9) | |
| H5 | 0.5968 | 0.3203 | 0.7526 | 0.061* | |
| C6 | 0.6426 (3) | 0.1889 (2) | 0.79315 (14) | 0.0392 (8) | |
| C7 | 0.8062 (3) | 0.0962 (2) | 0.83803 (15) | 0.0407 (8) | |
| C8 | 0.9449 (3) | 0.0627 (2) | 0.86263 (16) | 0.0428 (8) | |
| H8A | 0.9477 | 0.0167 | 0.8970 | 0.051* | |
| H8B | 0.9806 | 0.0315 | 0.8292 | 0.051* | |
| C9 | 0.9971 (3) | 0.1834 (3) | 0.94789 (15) | 0.0483 (9) | |
| H9A | 0.9372 | 0.1376 | 0.9619 | 0.058* | |
| H9B | 1.0800 | 0.1825 | 0.9787 | 0.058* | |
| C10 | 0.9390 (3) | 0.2821 (2) | 0.94594 (14) | 0.0368 (7) | |
| C11 | 0.8612 (3) | 0.4177 (3) | 0.97900 (15) | 0.0424 (8) | |
| C12 | 0.8176 (3) | 0.4915 (3) | 1.01283 (17) | 0.0539 (9) | |
| H12 | 0.8151 | 0.4852 | 1.0556 | 0.065* | |
| C13 | 0.7781 (3) | 0.5753 (3) | 0.9795 (2) | 0.0586 (10) | |
| H13 | 0.7479 | 0.6271 | 1.0005 | 0.070* | |
| C14 | 0.7816 (3) | 0.5853 (3) | 0.91592 (18) | 0.0516 (9) | |
| H14 | 0.7555 | 0.6438 | 0.8955 | 0.062* | |
| C15 | 0.8229 (3) | 0.5102 (2) | 0.88240 (16) | 0.0451 (8) | |
| H15 | 0.8239 | 0.5166 | 0.8395 | 0.054* | |
| C16 | 0.8624 (3) | 0.4259 (2) | 0.91432 (15) | 0.0378 (7) | |
| C17 | 1.1664 (3) | 0.1394 (2) | 0.88752 (16) | 0.0412 (8) | |
| H17A | 1.2099 | 0.1126 | 0.9281 | 0.049* | |
| H17B | 1.1807 | 0.0956 | 0.8542 | 0.049* | |
| C18 | 1.2211 (3) | 0.2369 (2) | 0.87831 (14) | 0.0349 (7) | |
| C19 | 1.3560 (3) | 0.3620 (2) | 0.87884 (14) | 0.0402 (8) | |
| C20 | 1.4596 (3) | 0.4284 (3) | 0.88702 (17) | 0.0552 (10) | |
| H20 | 1.5444 | 0.4124 | 0.9089 | 0.066* | |
| C21 | 1.4307 (3) | 0.5183 (3) | 0.86141 (17) | 0.0568 (10) | |
| H21 | 1.4979 | 0.5645 | 0.8657 | 0.068* | |
| C22 | 1.3051 (3) | 0.5433 (3) | 0.82922 (16) | 0.0514 (9) | |
| H22 | 1.2898 | 0.6062 | 0.8136 | 0.062* | |
| C23 | 1.2022 (3) | 0.4777 (2) | 0.81965 (15) | 0.0421 (8) | |
| H23 | 1.1181 | 0.4944 | 0.7973 | 0.051* | |
| C24 | 1.2294 (3) | 0.3859 (2) | 0.84485 (14) | 0.0361 (7) | |
| O1W | 0.9939 (3) | 0.2955 (2) | 0.60165 (12) | 0.0715 (8) | |
| H1A | 1.073 (2) | 0.302 (4) | 0.599 (2) | 0.107* | |
| H1B | 0.984 (4) | 0.283 (4) | 0.6383 (12) | 0.107* | |
| Cl3 | 1.26515 (10) | 0.36822 (8) | 0.57345 (5) | 0.0690 (3) | |
| O1 | 0.5693 (3) | 0.2940 (2) | 0.48243 (13) | 0.0781 (9) | |
| C25 | 0.5735 (5) | 0.2366 (3) | 0.5376 (2) | 0.0854 (14) | |
| H25A | 0.6237 | 0.1776 | 0.5349 | 0.102* | |
| H25B | 0.4842 | 0.2184 | 0.5413 | 0.102* | |
| C26 | 0.6369 (5) | 0.2936 (4) | 0.5937 (2) | 0.0999 (17) | |
| H26A | 0.7268 | 0.2714 | 0.6098 | 0.120* | 0.58 |
| H26B | 0.5867 | 0.2888 | 0.6272 | 0.120* | 0.58 |
| H26C | 0.7138 | 0.2598 | 0.6174 | 0.120* | 0.42 |
| H26D | 0.5754 | 0.3058 | 0.6215 | 0.120* | 0.42 |

| | | | | | |
|---|---|---|---|---|---|
| C27 | 0.6360 (17) | 0.3975 (6) | 0.5690 (5) | 0.088 (6) | 0.58 |
| H27A | 0.7114 | 0.4345 | 0.5913 | 0.106* | 0.58 |
| H27B | 0.5545 | 0.4312 | 0.5717 | 0.106* | 0.58 |
| C28 | 0.6462 (10) | 0.3772 (7) | 0.4990 (5) | 0.084 (4) | 0.58 |
| H28A | 0.6115 | 0.4314 | 0.4719 | 0.101* | 0.58 |
| H28B | 0.7376 | 0.3657 | 0.4956 | 0.101* | 0.58 |
| C28′ | 0.5865 (15) | 0.3944 (7) | 0.4996 (6) | 0.085 (5) | 0.42 |
| H28C | 0.5024 | 0.4247 | 0.5018 | 0.102* | 0.42 |
| H28D | 0.6277 | 0.4299 | 0.4698 | 0.102* | 0.42 |
| C27′ | 0.679 (2) | 0.3894 (9) | 0.5661 (6) | 0.084 (7) | 0.42 |
| H27C | 0.7717 | 0.3873 | 0.5626 | 0.101* | 0.42 |
| H27D | 0.6653 | 0.4447 | 0.5920 | 0.101* | 0.42 |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Fe1 | 0.0333 (2) | 0.0318 (3) | 0.0293 (2) | 0.00082 (18) | 0.00167 (17) | 0.00084 (19) |
| Cl1 | 0.0463 (4) | 0.0451 (5) | 0.0424 (5) | 0.0001 (4) | 0.0096 (3) | −0.0090 (4) |
| Cl2 | 0.0550 (5) | 0.0437 (5) | 0.0397 (5) | 0.0064 (4) | −0.0010 (4) | 0.0092 (4) |
| N1 | 0.0365 (14) | 0.0339 (16) | 0.0391 (15) | −0.0016 (11) | 0.0046 (11) | 0.0021 (12) |
| N2 | 0.0503 (17) | 0.0416 (18) | 0.0534 (19) | −0.0107 (15) | 0.0116 (14) | 0.0037 (15) |
| N3 | 0.0415 (14) | 0.0363 (16) | 0.0308 (14) | 0.0033 (11) | 0.0037 (11) | −0.0003 (12) |
| N4 | 0.0504 (16) | 0.052 (2) | 0.0296 (15) | 0.0020 (14) | 0.0084 (13) | 0.0017 (14) |
| N5 | 0.0340 (13) | 0.0291 (14) | 0.0363 (15) | −0.0007 (11) | −0.0006 (11) | −0.0004 (12) |
| N6 | 0.0359 (15) | 0.0472 (19) | 0.0453 (17) | 0.0058 (13) | −0.0050 (12) | 0.0021 (14) |
| N7 | 0.0405 (14) | 0.0316 (15) | 0.0348 (14) | 0.0048 (11) | 0.0041 (11) | 0.0013 (12) |
| C1 | 0.0414 (18) | 0.055 (2) | 0.0401 (19) | −0.0032 (16) | 0.0109 (15) | −0.0033 (17) |
| C2 | 0.049 (2) | 0.078 (3) | 0.058 (2) | −0.020 (2) | 0.0176 (18) | −0.010 (2) |
| C3 | 0.0372 (19) | 0.098 (3) | 0.056 (2) | −0.004 (2) | 0.0101 (17) | −0.018 (2) |
| C4 | 0.042 (2) | 0.077 (3) | 0.057 (2) | 0.0144 (19) | 0.0026 (17) | 0.000 (2) |
| C5 | 0.049 (2) | 0.054 (2) | 0.050 (2) | 0.0040 (17) | 0.0093 (17) | −0.0025 (18) |
| C6 | 0.0365 (17) | 0.047 (2) | 0.0348 (18) | 0.0014 (15) | 0.0079 (14) | −0.0036 (15) |
| C7 | 0.0407 (17) | 0.040 (2) | 0.0416 (19) | −0.0035 (15) | 0.0084 (14) | −0.0015 (16) |
| C8 | 0.0458 (18) | 0.0325 (19) | 0.048 (2) | 0.0013 (14) | 0.0037 (15) | 0.0033 (16) |
| C9 | 0.061 (2) | 0.047 (2) | 0.0361 (19) | 0.0098 (17) | 0.0095 (16) | 0.0042 (16) |
| C10 | 0.0384 (16) | 0.040 (2) | 0.0309 (17) | −0.0004 (14) | 0.0047 (13) | 0.0000 (15) |
| C11 | 0.0401 (17) | 0.044 (2) | 0.043 (2) | −0.0008 (15) | 0.0082 (15) | −0.0065 (16) |
| C12 | 0.057 (2) | 0.058 (3) | 0.050 (2) | −0.0008 (18) | 0.0179 (17) | −0.016 (2) |
| C13 | 0.056 (2) | 0.049 (2) | 0.074 (3) | 0.0028 (18) | 0.0196 (19) | −0.023 (2) |
| C14 | 0.0475 (19) | 0.043 (2) | 0.062 (2) | 0.0049 (16) | 0.0045 (17) | −0.0037 (19) |
| C15 | 0.0487 (19) | 0.037 (2) | 0.048 (2) | 0.0072 (15) | 0.0054 (15) | −0.0022 (17) |
| C16 | 0.0340 (16) | 0.040 (2) | 0.0391 (19) | 0.0007 (14) | 0.0059 (13) | −0.0056 (15) |
| C17 | 0.0386 (17) | 0.0341 (19) | 0.048 (2) | 0.0068 (14) | 0.0005 (14) | 0.0033 (16) |
| C18 | 0.0360 (16) | 0.0300 (18) | 0.0352 (17) | 0.0039 (13) | −0.0010 (13) | −0.0027 (14) |
| C19 | 0.0375 (17) | 0.041 (2) | 0.0401 (19) | −0.0015 (14) | 0.0025 (14) | −0.0003 (16) |
| C20 | 0.0357 (18) | 0.070 (3) | 0.054 (2) | −0.0071 (17) | −0.0051 (16) | 0.000 (2) |
| C21 | 0.051 (2) | 0.056 (3) | 0.061 (2) | −0.0186 (18) | 0.0048 (18) | 0.004 (2) |
| C22 | 0.058 (2) | 0.047 (2) | 0.048 (2) | −0.0103 (17) | 0.0088 (17) | 0.0069 (18) |

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|---|---|---|---|---|---|---|
| C23 | 0.0450 (18) | 0.042 (2) | 0.0383 (18) | −0.0008 (15) | 0.0056 (14) | 0.0053 (16) |
| C24 | 0.0380 (16) | 0.039 (2) | 0.0295 (16) | −0.0019 (14) | 0.0038 (13) | −0.0051 (14) |
| O1W | 0.082 (2) | 0.088 (2) | 0.0449 (16) | −0.0061 (18) | 0.0147 (14) | −0.0079 (16) |
| Cl3 | 0.0691 (6) | 0.0644 (7) | 0.0706 (7) | −0.0083 (5) | 0.0075 (5) | −0.0187 (5) |
| O1 | 0.081 (2) | 0.082 (2) | 0.0597 (19) | −0.0229 (17) | −0.0139 (14) | −0.0003 (17) |
| C25 | 0.083 (3) | 0.075 (3) | 0.093 (4) | −0.005 (2) | 0.006 (3) | 0.013 (3) |
| C26 | 0.102 (4) | 0.119 (5) | 0.072 (3) | −0.026 (3) | 0.002 (3) | −0.006 (3) |
| C27 | 0.083 (9) | 0.089 (10) | 0.086 (10) | −0.010 (7) | 0.001 (6) | −0.027 (7) |
| C28 | 0.072 (7) | 0.078 (7) | 0.105 (9) | −0.002 (5) | 0.024 (6) | 0.026 (6) |
| C28′ | 0.088 (12) | 0.079 (11) | 0.091 (11) | −0.024 (8) | 0.023 (9) | 0.000 (8) |
| C27′ | 0.087 (14) | 0.069 (11) | 0.080 (13) | 0.020 (7) | −0.021 (8) | −0.021 (8) |

*Geometric parameters (Å, º)*

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| Fe1—N5 | 2.097 (2) | C13—C14 | 1.384 (5) |
| Fe1—N1 | 2.100 (2) | C13—H13 | 0.9300 |
| Fe1—N3 | 2.107 (2) | C14—C15 | 1.375 (4) |
| Fe1—Cl2 | 2.2373 (9) | C14—H14 | 0.9300 |
| Fe1—Cl1 | 2.3219 (9) | C15—C16 | 1.367 (4) |
| Fe1—N7 | 2.355 (2) | C15—H15 | 0.9300 |
| Cl1—H1B | 2.51 (3) | C17—C18 | 1.482 (4) |
| N1—C7 | 1.335 (4) | C17—H17A | 0.9700 |
| N1—C6 | 1.405 (4) | C17—H17B | 0.9700 |
| N2—C7 | 1.333 (4) | C19—C20 | 1.387 (4) |
| N2—C1 | 1.382 (4) | C19—C24 | 1.399 (4) |
| N2—H2 | 0.859 (18) | C20—C21 | 1.361 (5) |
| N3—C10 | 1.325 (4) | C20—H20 | 0.9300 |
| N3—C16 | 1.422 (4) | C21—C22 | 1.381 (5) |
| N4—C10 | 1.338 (4) | C21—H21 | 0.9300 |
| N4—C11 | 1.388 (4) | C22—C23 | 1.375 (4) |
| N4—H4 | 0.862 (18) | C22—H22 | 0.9300 |
| N5—C18 | 1.320 (4) | C23—C24 | 1.379 (4) |
| N5—C24 | 1.402 (4) | C23—H23 | 0.9300 |
| N6—C18 | 1.343 (4) | O1W—H1A | 0.828 (18) |
| N6—C19 | 1.389 (4) | O1W—H1B | 0.831 (19) |
| N6—H6 | 0.852 (18) | Cl3—H1A | 2.34 (2) |
| N7—C8 | 1.484 (4) | O1—C28 | 1.395 (8) |
| N7—C17 | 1.484 (4) | O1—C25 | 1.420 (5) |
| N7—C9 | 1.486 (4) | O1—C28′ | 1.430 (9) |
| C1—C2 | 1.391 (4) | C25—C26 | 1.476 (5) |
| C1—C6 | 1.403 (4) | C25—H25A | 0.9700 |
| C2—C3 | 1.384 (5) | C25—H25B | 0.9700 |
| C2—H2A | 0.9300 | C26—C27 | 1.523 (8) |
| C3—C4 | 1.385 (6) | C26—C27′ | 1.544 (9) |
| C3—H3 | 0.9300 | C26—H26A | 0.9700 |
| C4—C5 | 1.386 (5) | C26—H26B | 0.9700 |
| C4—H4A | 0.9300 | C26—H26C | 0.9700 |
| C5—C6 | 1.374 (5) | C26—H26D | 0.9700 |

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| C5—H5 | 0.9300 | C27—C28 | 1.556 (9) |
| C7—C8 | 1.492 (4) | C27—H27A | 0.9700 |
| C8—H8A | 0.9700 | C27—H27B | 0.9700 |
| C8—H8B | 0.9700 | C28—H28A | 0.9700 |
| C9—C10 | 1.479 (4) | C28—H28B | 0.9700 |
| C9—H9A | 0.9700 | C28′—C27′ | 1.553 (10) |
| C9—H9B | 0.9700 | C28′—H28C | 0.9700 |
| C11—C12 | 1.376 (5) | C28′—H28D | 0.9700 |
| C11—C16 | 1.400 (4) | C27′—H27C | 0.9700 |
| C12—C13 | 1.374 (5) | C27′—H27D | 0.9700 |
| C12—H12 | 0.9300 | | |

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| N5—Fe1—N1 | 148.56 (10) | C15—C14—H14 | 119.4 |
| N5—Fe1—N3 | 85.80 (9) | C13—C14—H14 | 119.4 |
| N1—Fe1—N3 | 86.65 (10) | C16—C15—C14 | 117.7 (3) |
| N5—Fe1—Cl2 | 106.78 (7) | C16—C15—H15 | 121.1 |
| N1—Fe1—Cl2 | 104.43 (7) | C14—C15—H15 | 121.1 |
| N3—Fe1—Cl2 | 97.23 (7) | C15—C16—C11 | 120.4 (3) |
| N5—Fe1—Cl1 | 89.19 (7) | C15—C16—N3 | 131.7 (3) |
| N1—Fe1—Cl1 | 91.09 (7) | C11—C16—N3 | 107.9 (3) |
| N3—Fe1—Cl1 | 166.40 (7) | C18—C17—N7 | 107.5 (2) |
| Cl2—Fe1—Cl1 | 96.32 (3) | C18—C17—H17A | 110.2 |
| N5—Fe1—N7 | 74.41 (9) | N7—C17—H17A | 110.2 |
| N1—Fe1—N7 | 74.16 (9) | C18—C17—H17B | 110.2 |
| N3—Fe1—N7 | 78.09 (9) | N7—C17—H17B | 110.2 |
| Cl2—Fe1—N7 | 175.13 (7) | H17A—C17—H17B | 108.5 |
| Cl1—Fe1—N7 | 88.40 (6) | N5—C18—N6 | 112.1 (3) |
| Fe1—Cl1—H1B | 103.7 (10) | N5—C18—C17 | 121.2 (3) |
| C7—N1—C6 | 105.1 (2) | N6—C18—C17 | 126.7 (3) |
| C7—N1—Fe1 | 116.72 (19) | C20—C19—N6 | 132.9 (3) |
| C6—N1—Fe1 | 138.1 (2) | C20—C19—C24 | 121.5 (3) |
| C7—N2—C1 | 107.7 (3) | N6—C19—C24 | 105.6 (3) |
| C7—N2—H2 | 125 (2) | C21—C20—C19 | 116.5 (3) |
| C1—N2—H2 | 127 (2) | C21—C20—H20 | 121.8 |
| C10—N3—C16 | 105.4 (2) | C19—C20—H20 | 121.8 |
| C10—N3—Fe1 | 116.1 (2) | C20—C21—C22 | 122.4 (3) |
| C16—N3—Fe1 | 138.5 (2) | C20—C21—H21 | 118.8 |
| C10—N4—C11 | 107.9 (3) | C22—C21—H21 | 118.8 |
| C10—N4—H4 | 120 (2) | C23—C22—C21 | 121.8 (3) |
| C11—N4—H4 | 132 (2) | C23—C22—H22 | 119.1 |
| C18—N5—C24 | 106.3 (2) | C21—C22—H22 | 119.1 |
| C18—N5—Fe1 | 118.0 (2) | C22—C23—C24 | 116.8 (3) |
| C24—N5—Fe1 | 135.7 (2) | C22—C23—H23 | 121.6 |
| C18—N6—C19 | 107.9 (2) | C24—C23—H23 | 121.6 |
| C18—N6—H6 | 130 (2) | C23—C24—C19 | 121.0 (3) |
| C19—N6—H6 | 122 (2) | C23—C24—N5 | 130.9 (3) |
| C8—N7—C17 | 112.7 (2) | C19—C24—N5 | 108.1 (3) |
| C8—N7—C9 | 111.3 (2) | H1A—O1W—H1B | 113 (3) |

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| C17—N7—C9 | 111.2 (2) | C28—O1—C25 | 108.8 (5) |
| C8—N7—Fe1 | 105.93 (17) | C25—O1—C28′ | 109.8 (6) |
| C17—N7—Fe1 | 106.90 (17) | C28—O1—H4A | 93.5 |
| C9—N7—Fe1 | 108.49 (18) | O1—C25—C26 | 108.8 (3) |
| N2—C1—C2 | 131.9 (3) | O1—C25—H25A | 109.9 |
| N2—C1—C6 | 106.0 (3) | C26—C25—H25A | 109.9 |
| C2—C1—C6 | 122.0 (3) | O1—C25—H25B | 109.9 |
| C3—C2—C1 | 115.6 (4) | C26—C25—H25B | 109.9 |
| C3—C2—H2A | 122.2 | H25A—C25—H25B | 108.3 |
| C1—C2—H2A | 122.2 | C25—C26—C27 | 104.0 (5) |
| C2—C3—C4 | 122.7 (3) | C25—C26—C27′ | 104.5 (6) |
| C2—C3—H3 | 118.6 | C25—C26—H26A | 111.0 |
| C4—C3—H3 | 118.6 | C27—C26—H26A | 111.0 |
| C3—C4—C5 | 121.1 (4) | C27′—C26—H26A | 94.8 |
| C3—C4—H4A | 119.4 | C25—C26—H26B | 111.0 |
| C5—C4—H4A | 119.4 | C27—C26—H26B | 111.0 |
| C6—C5—C4 | 117.4 (4) | C27′—C26—H26B | 125.4 |
| C6—C5—H5 | 121.3 | H26A—C26—H26B | 109.0 |
| C4—C5—H5 | 121.3 | C25—C26—H26C | 110.8 |
| C5—C6—C1 | 121.1 (3) | C27—C26—H26C | 125.4 |
| C5—C6—N1 | 130.7 (3) | C27′—C26—H26C | 110.4 |
| C1—C6—N1 | 108.2 (3) | H26B—C26—H26C | 94.3 |
| N2—C7—N1 | 112.9 (3) | C25—C26—H26D | 111.2 |
| N2—C7—C8 | 126.0 (3) | C27′—C26—H26D | 111.1 |
| N1—C7—C8 | 121.1 (3) | H26A—C26—H26D | 121.8 |
| N7—C8—C7 | 106.0 (2) | H26C—C26—H26D | 108.7 |
| N7—C8—H8A | 110.5 | C26—C27—C28 | 100.0 (7) |
| C7—C8—H8A | 110.5 | C26—C27—H27A | 111.8 |
| N7—C8—H8B | 110.5 | C28—C27—H27A | 111.8 |
| C7—C8—H8B | 110.5 | C26—C27—H27B | 111.8 |
| H8A—C8—H8B | 108.7 | C28—C27—H27B | 111.8 |
| C10—C9—N7 | 112.9 (3) | H27A—C27—H27B | 109.5 |
| C10—C9—H9A | 109.0 | O1—C28—C27 | 104.4 (7) |
| N7—C9—H9A | 109.0 | O1—C28—H28A | 110.9 |
| C10—C9—H9B | 109.0 | C27—C28—H28A | 110.9 |
| N7—C9—H9B | 109.0 | O1—C28—H28B | 110.9 |
| H9A—C9—H9B | 107.8 | C27—C28—H28B | 110.9 |
| N3—C10—N4 | 112.9 (3) | H28A—C28—H28B | 108.9 |
| N3—C10—C9 | 124.3 (3) | O1—C28′—C27′ | 102.5 (8) |
| N4—C10—C9 | 122.9 (3) | O1—C28′—H28C | 111.3 |
| C12—C11—N4 | 131.5 (3) | C27′—C28′—H28C | 111.3 |
| C12—C11—C16 | 122.5 (3) | O1—C28′—H28D | 111.3 |
| N4—C11—C16 | 106.0 (3) | C27′—C28′—H28D | 111.3 |
| C13—C12—C11 | 115.9 (3) | H28C—C28′—H28D | 109.2 |
| C13—C12—H12 | 122.1 | C26—C27′—C28′ | 102.9 (8) |
| C11—C12—H12 | 122.1 | C26—C27′—H27C | 111.2 |
| C12—C13—C14 | 122.4 (3) | C28′—C27′—H27C | 111.2 |
| C12—C13—H13 | 118.8 | C26—C27′—H27D | 111.2 |

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| C14—C13—H13 | 118.8 | C28′—C27′—H27D | 111.2 |
| C15—C14—C13 | 121.1 (4) | H27C—C27′—H27D | 109.1 |
| | | | |
| N5—Fe1—Cl1—H1B | −96.2 (10) | N1—C7—C8—N7 | 30.8 (4) |
| N1—Fe1—Cl1—H1B | 115.2 (10) | C8—N7—C9—C10 | −118.9 (3) |
| N3—Fe1—Cl1—H1B | −164.5 (10) | C17—N7—C9—C10 | 114.5 (3) |
| Cl2—Fe1—Cl1—H1B | 10.6 (10) | Fe1—N7—C9—C10 | −2.7 (3) |
| N7—Fe1—Cl1—H1B | −170.7 (10) | C16—N3—C10—N4 | 1.5 (3) |
| N5—Fe1—N1—C7 | −20.2 (3) | Fe1—N3—C10—N4 | −177.65 (19) |
| N3—Fe1—N1—C7 | −96.6 (2) | C16—N3—C10—C9 | −177.9 (3) |
| Cl2—Fe1—N1—C7 | 166.8 (2) | Fe1—N3—C10—C9 | 2.9 (4) |
| Cl1—Fe1—N1—C7 | 70.0 (2) | C11—N4—C10—N3 | −1.2 (3) |
| N7—Fe1—N1—C7 | −18.0 (2) | C11—N4—C10—C9 | 178.3 (3) |
| N5—Fe1—N1—C6 | 161.4 (3) | N7—C9—C10—N3 | 0.2 (5) |
| N3—Fe1—N1—C6 | 85.1 (3) | N7—C9—C10—N4 | −179.2 (3) |
| Cl2—Fe1—N1—C6 | −11.6 (3) | C10—N4—C11—C12 | −179.8 (3) |
| Cl1—Fe1—N1—C6 | −108.4 (3) | C10—N4—C11—C16 | 0.4 (3) |
| N7—Fe1—N1—C6 | 163.6 (3) | N4—C11—C12—C13 | 178.6 (3) |
| N5—Fe1—N3—C10 | −78.2 (2) | C16—C11—C12—C13 | −1.5 (5) |
| N1—Fe1—N3—C10 | 71.3 (2) | C11—C12—C13—C14 | 0.0 (5) |
| Cl2—Fe1—N3—C10 | 175.4 (2) | C12—C13—C14—C15 | 1.3 (5) |
| Cl1—Fe1—N3—C10 | −9.5 (4) | C13—C14—C15—C16 | −1.0 (5) |
| N7—Fe1—N3—C10 | −3.2 (2) | C14—C15—C16—C11 | −0.5 (5) |
| N5—Fe1—N3—C16 | 103.0 (3) | C14—C15—C16—N3 | −179.0 (3) |
| N1—Fe1—N3—C16 | −107.5 (3) | C12—C11—C16—C15 | 1.8 (5) |
| Cl2—Fe1—N3—C16 | −3.4 (3) | N4—C11—C16—C15 | −178.3 (3) |
| Cl1—Fe1—N3—C16 | 171.7 (2) | C12—C11—C16—N3 | −179.3 (3) |
| N7—Fe1—N3—C16 | 178.0 (3) | N4—C11—C16—N3 | 0.5 (3) |
| N1—Fe1—N5—C18 | 16.6 (3) | C10—N3—C16—C15 | 177.4 (3) |
| N3—Fe1—N5—C18 | 93.2 (2) | Fe1—N3—C16—C15 | −3.7 (5) |
| Cl2—Fe1—N5—C18 | −170.5 (2) | C10—N3—C16—C11 | −1.2 (3) |
| Cl1—Fe1—N5—C18 | −74.2 (2) | Fe1—N3—C16—C11 | 177.6 (2) |
| N7—Fe1—N5—C18 | 14.4 (2) | C8—N7—C17—C18 | 151.7 (3) |
| N1—Fe1—N5—C24 | −161.7 (2) | C9—N7—C17—C18 | −82.5 (3) |
| N3—Fe1—N5—C24 | −85.1 (3) | Fe1—N7—C17—C18 | 35.7 (3) |
| Cl2—Fe1—N5—C24 | 11.2 (3) | C24—N5—C18—N6 | −0.2 (3) |
| Cl1—Fe1—N5—C24 | 107.6 (3) | Fe1—N5—C18—N6 | −178.9 (2) |
| N7—Fe1—N5—C24 | −163.9 (3) | C24—N5—C18—C17 | −178.6 (3) |
| N5—Fe1—N7—C8 | −148.3 (2) | Fe1—N5—C18—C17 | 2.7 (4) |
| N1—Fe1—N7—C8 | 32.90 (18) | C19—N6—C18—N5 | 0.2 (4) |
| N3—Fe1—N7—C8 | 122.78 (19) | C19—N6—C18—C17 | 178.5 (3) |
| Cl1—Fe1—N7—C8 | −58.69 (18) | N7—C17—C18—N5 | −28.4 (4) |
| N5—Fe1—N7—C17 | −27.88 (18) | N7—C17—C18—N6 | 153.5 (3) |
| N1—Fe1—N7—C17 | 153.3 (2) | C18—N6—C19—C20 | 179.1 (4) |
| N3—Fe1—N7—C17 | −116.80 (19) | C18—N6—C19—C24 | −0.2 (3) |
| Cl1—Fe1—N7—C17 | 61.73 (17) | N6—C19—C20—C21 | −177.9 (4) |
| N5—Fe1—N7—C9 | 92.1 (2) | C24—C19—C20—C21 | 1.3 (5) |
| N1—Fe1—N7—C9 | −86.7 (2) | C19—C20—C21—C22 | 0.4 (6) |

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| N3—Fe1—N7—C9 | 3.18 (19) | C20—C21—C22—C23 | −1.7 (6) |
| Cl1—Fe1—N7—C9 | −178.29 (19) | C21—C22—C23—C24 | 1.2 (5) |
| C7—N2—C1—C2 | 176.9 (3) | C22—C23—C24—C19 | 0.5 (5) |
| C7—N2—C1—C6 | −1.2 (4) | C22—C23—C24—N5 | 177.4 (3) |
| N2—C1—C2—C3 | −177.6 (3) | C20—C19—C24—C23 | −1.8 (5) |
| C6—C1—C2—C3 | 0.3 (5) | N6—C19—C24—C23 | 177.6 (3) |
| C1—C2—C3—C4 | −0.6 (5) | C20—C19—C24—N5 | −179.3 (3) |
| C2—C3—C4—C5 | 0.4 (6) | N6—C19—C24—N5 | 0.1 (3) |
| C3—C4—C5—C6 | 0.0 (5) | C18—N5—C24—C23 | −177.2 (3) |
| C4—C5—C6—C1 | −0.3 (5) | Fe1—N5—C24—C23 | 1.3 (5) |
| C4—C5—C6—N1 | 176.4 (3) | C18—N5—C24—C19 | 0.0 (3) |
| N2—C1—C6—C5 | 178.5 (3) | Fe1—N5—C24—C19 | 178.5 (2) |
| C2—C1—C6—C5 | 0.1 (5) | C28—O1—C25—C26 | 10.0 (7) |
| N2—C1—C6—N1 | 1.2 (3) | C28′—O1—C25—C26 | −18.6 (8) |
| C2—C1—C6—N1 | −177.2 (3) | H4A—O1—C25—C26 | −61.4 |
| C7—N1—C6—C5 | −177.6 (3) | O1—C25—C26—C27 | 14.7 (8) |
| Fe1—N1—C6—C5 | 0.9 (5) | O1—C25—C26—C27′ | −3.7 (11) |
| C7—N1—C6—C1 | −0.6 (3) | C25—C26—C27—C28 | −30.5 (10) |
| Fe1—N1—C6—C1 | 177.9 (2) | C27′—C26—C27—C28 | 64 (2) |
| C1—N2—C7—N1 | 0.9 (4) | C25—O1—C28—C27 | −30.0 (10) |
| C1—N2—C7—C8 | −177.3 (3) | C28′—O1—C28—C27 | 67.0 (18) |
| C6—N1—C7—N2 | −0.1 (4) | H4A—O1—C28—C27 | 6.6 |
| Fe1—N1—C7—N2 | −179.0 (2) | C26—C27—C28—O1 | 37.2 (11) |
| C6—N1—C7—C8 | 178.1 (3) | C28—O1—C28′—C27′ | −60.6 (18) |
| Fe1—N1—C7—C8 | −0.8 (4) | C25—O1—C28′—C27′ | 32.1 (13) |
| C17—N7—C8—C7 | −157.2 (2) | H4A—O1—C28′—C27′ | 57.8 |
| C9—N7—C8—C7 | 77.1 (3) | C25—C26—C27′—C28′ | 22.1 (15) |
| Fe1—N7—C8—C7 | −40.6 (3) | C27—C26—C27′—C28′ | −68 (2) |
| N2—C7—C8—N7 | −151.2 (3) | O1—C28′—C27′—C26 | −32.6 (16) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1*W*—H1*A*···Cl3 | 0.83 (2) | 2.34 (2) | 3.136 (3) | 160 (4) |
| O1*W*—H1*B*···Cl1 | 0.83 (2) | 2.51 (3) | 3.267 (3) | 152 (4) |
| C23—H23···Cl1$^{i}$ | 0.93 | 2.82 | 3.516 (3) | 133 |
| N2—H2···Cl3$^{ii}$ | 0.86 (2) | 2.25 (2) | 3.070 (3) | 159 (3) |
| N4—H4···O1*W*$^{iii}$ | 0.86 (2) | 1.96 (2) | 2.795 (4) | 162 (3) |
| N6—H6···O1$^{iv}$ | 0.85 (2) | 1.91 (2) | 2.741 (4) | 163 (3) |
| C9—H9*B*···Cl3$^{iii}$ | 0.97 | 2.59 | 3.523 (3) | 162 |
| C3—H3···Cl1$^{v}$ | 0.93 | 2.83 | 3.718 (4) | 160 |
| C15—H15···Cl2 | 0.93 | 2.74 | 3.466 (3) | 136 |

Symmetry codes: (i) −*x*+2, *y*+1/2, −*z*+3/2; (ii) −*x*+2, *y*−1/2, −*z*+3/2; (iii) *x*, −*y*+1/2, *z*+1/2; (iv) *x*+1, −*y*+1/2, *z*+1/2; (v) *x*−1, *y*, *z*.